



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : D04H 1/64, D06M 10/00		A1	(11) International Publication Number: WO 95/35402
			(43) International Publication Date: 28 December 1995 (28.12.95)
(21) International Application Number: PCT/EP95/02325		(81) Designated States: AU, BR, CA, CN, CZ, FI, HU, JP, KR, NO, NZ, PL, RU, SI, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).	
(22) International Filing Date: 16 June 1995 (16.06.95)			
(30) Priority Data: P 44 21 254.2 17 June 1994 (17.06.94) DE		Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.	
(71) Applicant (for all designated States except US): ISOVER SAINT-GOBAIN [FR/FR]; 18, avenue d'Alsace, F-92400 Courbevoie (FR).			
(72) Inventors; and (75) Inventors/Applicants (for US only): MERTZ, Frédéric [FR/FR]; 16, rue Marcel-Sembat, F-93350 Le Bourget (FR). CHARTIER, Pascal [FR/FR]; Bâtiment 6, 45, boulevard Dubrenil, F-91400 Orsay (FR). DECKER, Christian [FR/FR]; 3, rue de l'Abattoir, F-68170 Rixheim (FR).			
(74) Agent: KUHNEN, WACKER & PARTNER; Alois-Steinecker-Strasse 22, D-85354 Freising (DE).			
(54) Title: METHOD AND DEVICE FOR POLYMERISING BINDERS IN FIBROUS MATERIALS ESPECIALLY MINERAL WOOL INSULATION			
(57) Abstract			
<p>The invention concerns a method and a device for polymerising substances in fibrous materials, in particular binding agents in mineral wool materials for insulation purposes, wherein the fibrous material having a given thickness (d) is subjected to UV irradiation in order to polymerise the substance having the form of a prepolymer impregnating the fibres, said irradiation of the material is carried out at a sufficiently high intensity level (I_{UV}) to bring about, at a maximum depth (d) of intended polymerisation, a residual intensity exceeding such threshold value at which polymerisation of the selected binding agent under the influence of UV radiation is ensured within a given time limit, such time limit, however, precluding any undesirable degradation, due to the effects of the radiation, of organic substances in the portion at the surface of said material, and wherein the duration of irradiating a given surface unit of said material is kept within said time limit.</p>			

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

Description

METHOD AND DEVICE FOR POLYMERISING BINDERS IN FIBROUS MATERIALS ESPECIALLY MINERAL WOOL INSULATION

The invention concerns a method for polymerising substances in fibrous materials, in particular binding agents in mineral wool materials for insulation purposes, wherein the fibrous material which has a given thickness and which, in a given case, may be continuously moving in a line of production, is subjected to UV irradiation in order to polymerise the substance having the form of a prepolymer impregnating the fibres, and a device for polymerising substances in fibrous materials, such as particularly a binding agent in a mineral wool material for insulation purposes, comprising at least one UV radiation source.

Such a technique is known from US-A-5 275 874. It is the purpose of the known teaching to uniformly polymerise the material across its thickness. In view of the fact that the energy released inside the material by the UV rays exponentially declines with increasing distance from the material surface, a considerable treatment duration is required in order to achieve maximum uniformity of curing. In the exemplary case of US-A-5 275 874, radiation intensity across the thickness of the material drops to 1% of the incident radiation as the material passes under a plurality of consecutively arranged UV lamps, essentially in analogy with a passage through the curing furnace in conventional thermal curing.

With such a treatment, experience within the framework of the present invention teaches that the

surface-side organic constituents of the mineral wool material are inevitably scorched or carbonised. In view of the rapid drop in intensity, i.e. the power released per surface unit, with the depth below the irradiated surface, the required energy input is so high as to bring about carbonising or scorching of the surface-side organic constituents, e.g. the binding agent, at the irradiated surface of the mineral wool material.

Correspondingly, the invention is based on the object of providing a method according to the preamble of claim 1 whereby the desired polymerisation is made possible even in considerable material depths below the irradiated surface without the occurrence of any undesirable decomposition effects at the material surface.

This object is achieved in that irradiation is carried out at a sufficiently high intensity level to bring about, at a maximum depth of intended polymerisation, a residual intensity exceeding such threshold value at which polymerisation of the selected binding agent under the influence of UV radiation is ensured within a given time limit, such time limit, however, precluding any undesirable degradation due to the effects of the radiation of organic substances in the portion at the surface of the material, and in that the duration of irradiating a surface unit of the material is kept within this time limit. In the case of a fibrous material continuously moving in a production line, this duration will also be selected so as to be compatible with the velocity of the production line.

In the course of research of the work relating to the present invention it was found that the materials eligible as a prepolymer exhibit threshold values for the intensity level, i.e. the irradiated power per area unit,

above which polymerisation takes place within a very short time period, virtually instantaneously. Increasing the intensity level above this threshold value does not appreciably accelerate polymerisation, without, however, unfavorably affecting it. The temperature increase at the irradiated surface leading to the occurrence of degradation effects, on the other hand, is essentially proportional to the duration of irradiation, with the velocity of the temperature increase naturally increasing as a function of the levels of intensity.

By the application of UV radiation, heat is released as a function of the radiation intensity, either due to the heat radiation concurrently emitted by the UV source, or due to energy losses of the UV radiation which is thus transformed into thermal radiation; these effects appear most clearly at the surface of the material, with the highest temperatures occurring there and possibly bringing about decomposition effects once a given temperature limit is exceeded.

These effects which occur in parallel, however with a different course over time, are used in the present invention in that firstly, the threshold level of intensity is determined at which polymerisation of the material occurs rapidly, e.g. within 0.2 s. The area unit in question is then irradiated for this duration, and the surface intensity level of irradiation may be raised to such an extent that the rise of temperature during this predetermined time span of e.g. 0.2 s still remains below a given temperature limit above which degradation effects have to be expected. This maximum intensity, which may be applied within the predetermined short period of time, due to the intensity drop inside the material, in turn limits the depth within which the intensity remains above

the threshold value and within which a virtually homogeneous polymerisation is therefore possible.

As each area unit is consequently exposed to a high
5 intensity level radiation for a short time period only, irradiation may be carried out in a limited space to save production line space. In the case of a continuously moving web of material in the production line, the time span of irradiation of each area unit has to be selected
10 so as to be compatible with the velocity of the line, generally between 0.1 and 1 m/s.

If the intensity level of irradiation is in excess of 500 mW/cm², in particular in excess of 1 W/cm², and in a
15 particularly preferred manner exceeds 2 W/cm², then commercially available UV sources may be applied for cost efficiency on the one hand while nevertheless enabling high surface intensity levels on the other hand. As is known *per se*, UV sources for radiation in the wavelength
20 area above 250 nm, preferably above 310 nm are preferred in view of the selectively elevated transmissivity of the mineral wool material in this area.

The duration of irradiating a surface unit is preferably
25 shorter than ten seconds, preferably shorter than 1 s, in particular shorter than 0.5 s.

In many conventional production methods, the mineral fibres are produced from molten glass, provided with the
30 substance, e.g. the prepolymer of the binding agent, in the course of deposition as a layer of wool, and then the substance, e.g. the binding agent, is cured. In such a case, the mineral wool material still has a relatively elevated temperature when the substance is being cured,
35 such that the temperature rise on the surface side to the temperature limit under the influence of the irradiation

starts out from a relatively high initial temperature. Particularly in such a case it is preferred to cool the mineral wool material to a temperature as low as possible before irradiation to thus allow for a larger permissible increase of temperature due to the irradiation treatment. Cooling measures concurrent with irradiation, however, in view of the short time period of irradiation do not have an effect that would justify the expenditure associated therewith. Applying the method of the invention, on the other hand, is particularly advantageous with all those production methods where the mineral wool material is present at a lower temperature, preferably at ambient temperature, before curing. This is the case e.g. when the binding agent is introduced in the form of vapor or of an aerosol, as is known from DE-A-44 06 863 and DE-A-44 10 020, respectively.

The proportion of oxygen contained in the fibrous material, which in usual processing is at about 21% in the surrounding atmosphere, is preferably reduced during irradiation to less than 10%, better even 5%, and in a particularly preferred manner to less than 1%. In usual polymerisation making use of radicals, this measure prevents the free radicals of the substance to be polymerised, which are generated by the radiation, from being occupied by oxygen and thus prevents a failure of polymerisation in those places. This is particularly important with very thin film thicknesses of monomers and low intensity of irradiation.

Irradiation is preferably applied to both major surfaces of the material, such that the radiation energy penetrates across the thickness of the material from both sides. The penetrating depth of the radiation may thus be more than doubled when both penetrating depths overlap in the centre of the material. This overlapping effect is

made particularly good use of if both sides are irradiated concurrently, such that in a certain location inside the material the energies from irradiating both sides are present simultaneously.

5

Polymerisation or curing does not necessarily have to be homogeneous across the thickness of the material. Especially in the case of bilateral irradiation, it may be perfectly sufficient if the layers of material adjacent to the surface are cured and a more or less large inner width of considerably reduced polymerisation remains. In the case of curing a binding agent, the desired increase in mechanical strength is thus effected in the outer portions, such that resistance against bending, which frequently is of primary importance, hardly suffers from a lower mechanical strength in the central portion of the panel. Incomplete curing is frequently also acceptable where other properties than the bending resistance, e.g. surface tightness, surface strength, or the like are of importance. When other substances than binding agents are polymerised, non-homogeneous polymerisation across the thickness may prove to be favourable depending on the function of the substance and the use of the product.

25

In the case of non-homogeneous polymerisation across the thickness it may also be favourable, in accordance with the teachings of DE-A-44 06 863 or DE-A-44 10 020, to already introduce the substance into the material in a non-homogeneous distribution. E.g. in the case of binding agent, a higher binding agent concentration may thereby be achieved in the portions near the surfaces than in the central portion of the panel, resulting in a production with a desired optimum use of a certain amount of binding agent.

35

For the prepolymer, in particular multi-functional acrylic and metacrylic compositions are suited, as is known *per se*. Further usable substances are prepolymers in the form of monomers, oligomers, or polymers comprising polymerisable, unsaturated functional groups such as acrylate, methacrylate, vinyl, vinyl ether, allylic, or maleate groups which are capable of reacting in the sense of a chain extension and/or cross-linking. The binding agent may be a mixture from these compositions and contains a photo-initiator in order to enable polymerisation through UV light. Prepolymer mixtures comprising epoxy acrylate were found to be particularly preferred with the present invention.

A device according to the invention serving to implement the method of the invention is characterised in that means for focussing the UV radiation to at least one focussed beam of small width are provided.

Due to such focussing, a desired high level of surface intensity may be achieved.

Particularly in polymersation at a running production line it may be preferred if focussing is effected in a linear shape, e.g. extending across the width of the production web. The width of the linear focussed beam may then be coordinated with the transport velocity of the production web to result in a desired duration of irradiating each surface unit without the presence of any moved parts.

As an alternative, however, focussing may also be effected in the shape of a point, with the thin high-intensity beam thus formed sweeping the material in a desired predetermined pattern. The substance may thus be cured in fibrous material having an uneven surface in the case of

shaped articles, e.g. pipe sections having the shape of a hollow cylinder. This movement of the focussed beam may, for example, even be obtained at a quite limited expenditure by means of a sweeper mirror. The high intensity or power density of such a beam focussed in the shape of a point enables large penetrating depths of polymerisation for substances having particularly short polymerising times. Particularly, such sweeping movement with a thin beam allows a good adaptation to different line velocities. For certain cases of use, it may suffice to sweep only a fraction of the total surface area of the fibrous material with the beam and to allow for gaps in between, within which polymerisation is not necessary or does in fact occur as a distant effect of the adjacent irradiation.

In a preferred manner, the focussed beam is directed at a finite focal point, i.e. tapering into a point instead of consisting of parallel rays. Thanks to suitable focussing, the focal point may be positioned at a desired depth of the material so that a relatively higher intensity on a smaller surface is obtained there, even though attenuated by scattering at fibres located further upwards. Such a formation of the focussed beam is particularly suitable in the case of a line-shaped focussed beam.

Further details, features and advantages of the invention can be seen from the following description of embodiments by making reference to the drawing, wherein:

Fig. 1 shows a typical function of the intensity level versus the wall thickness of a mineral wool product subjected to bilateral UV irradiation,

35

Fig. 2 shows the rate of conversion by polymerisation versus time at various intensity levels,

Fig. 3 shows the surface heating of the mineral wool material at various intensity levels,

Fig. 4 shows the relation of intensity level and polymerisation across the wall thickness of a mineral wool product treated according to the invention with bilateral UV irradiation,

Fig. 5 shows a schematically simplified representation of a device according to the invention having linear focussing of the UV radiation, and

Fig. 6 shows a schematically simplified representation of a device according to the invention having a point-shaped focussed beam on the mineral wool material.

In order to facilitate comprehension, the same reference signs are used for identical or corresponding parts or graphically represented functions, respectively.

Fig. 1 illustrates a typical function of the local intensity level of UV radiation versus the thickness of a fibrous material, for example a mineral wool product subjected to bilateral UV irradiation of its surface.

As the abscissa or X axis, the wall thickness or depth d of the mineral wool product of, in the case of the example, a wall thickness of 12 cm is shown. The left ordinate or Y axis shows the intensity level of the UV radiation I_{UV} of 0 to 100% and the right ordinate or Y axis shows the intensity level of the UV radiation I_{UV} of 0 to 2000 mW/cm². In the example shown, 2000 mW/cm²

correspond to the 100% intensity level of the UV radiation.

On the right side of the right ordinate, on the one hand, and on the left side of the left ordinate, on the other hand, arrows are drawn which, pointing towards the middle of the Figure, symbolize the incident UV radiation having the energy $h\nu$. Since irradiation occurs from both sides of the mineral wool product, the intensity level over the thickness d illustrated by curve 1 is approximately symmetrical to the centre line so that initially only the left half of the diagram shall be considered herebelow. It can be seen that the intensity level I_{UV} sharply drops already after a relatively short penetration depth d . From an initial intensity level I_{UV} of 100% at the mineral wool surface, the intensity level I_{UV} drops, in this example, to below 50% already after less than 1 cm penetration depth, after a penetration depth of approximately 2 cm already to below 10% and, in the middle portion of the mineral wool product, amounts to only below 5% of the intensity level I_{UV} incident on the surface. This sharp decline of the intensity level I_{UV} over the penetration depth is, on the one hand, caused by the low transmission of the UV radiation through the mineral wool material and, on the other hand, a result of a conversion of UV radiation energy to thermal energy already in the uppermost layers of the mineral wool material, without a related polymerisation.

A first evaluation of curve 1 of intensity as shown leads to the result that a satisfactory polymerisation of substances in fibrous materials must be restricted to portions of material near the surfaces since the UV radiation intensity level I_{UV} decreases too rapidly, thus failing to provide a sufficiently high residual intensity in the centre portion for accomplishing satisfactory

polymerisation there. If, therefore, the intensity level I_{UV} of the radiation were increased to such an extent that the centre portion of the fibrous material can still be successfully polymerised, this would entail such high intensity levels at the surface that damage to the surface-side material, for example carbonisation or scorching of the surface-side organic constituents, e.g. the polymerisable substance or a binding agent, could not be avoided.

10

In Fig. 2 the function of the polymerisation of substances in fibrous materials versus the time at various intensity levels of the UV radiation is shown. At the abscissa or X axis, the irradiation time t in seconds is shown from 0 to 1.5 s. The left ordinate or Y axis shows the related conversion rate by polymerisation P from 0 to 100%. The curves I_{UV1} to I_{UV7} show various intensity levels I_{UV} of the UV radiation of, for example, $I_{UV1} = 2 \text{ mW/cm}^2$, $I_{UV2} = 5 \text{ mW/cm}^2$, $I_{UV3} = 11 \text{ mW/cm}^2$, $I_{UV4} = 25 \text{ mW/cm}^2$, $I_{UV5} = 55 \text{ mW/cm}^2$, $I_{UV6} = 128 \text{ mW/cm}^2$, and $I_{UV7} = 220 \text{ mW/cm}^2$. Thus, the intensity level I_{UV} increases from I_{UV1} to I_{UV7} .

All seven curves exhibit typical common features. Those are, firstly, a common point of origin in the origin 0 of the graph. After a short delay, this common point of origin is followed by an ascent of the curve with a positive gradient dP/dt , which is followed by an almost linear area of ascent with a constant positive gradient dP/dt . This area of linear ascent, furthermore, is followed in all shown curves by a decreasing ascent, i.e. having a decreasing positive gradient dP/dt , to arrive at an almost horizontal linear section without further ascent, i.e. having a constant gradient dP/dt of approximately 0. The limit of the conversion rate by polymerisation P reached in this constant section can be

designated as a saturation limit of the polymerisation P occurring at a specific intensity level I_{UV} of radiation.

The steep gradient of the linear ascents in the front
5 portion of the curves increases with increasing intensity
 I_{UV} of the UV radiation, and is not very distinct in the
case of the curve for I_{UV1} with 2 mW/cm², whereas a very
steep gradient is present for the curve I_{UV7} with
220 mW/cm². The arc of transition from the steep ascent
10 in the front portion of the curves to the horizontal,
almost linear portion of the curves is not very distinct
in the case of the two intensity levels I_{UV1} and I_{UV2} , in
the case of the intensity level I_{UV3} it is well
discernable, and the intensity levels I_{UV4} to I_{UV7} exhibit
15 a clearly distinct arc of transition from the sharp
ascents to the constant portions of the curves. Moreover
this arc of transition occurs at an earlier point in time
with increasing intensity level I_{UV} .

20 This means that, in the cases of lower intensity
levels I_{UV} of UV radiation, the mineral wool material has
to be irradiated for a relatively longer time until no
further increase of polymerisation, which is low at any
rate, occurs. However, if a relatively high UV radiation
25 is used for irradiating the mineral wool material, a high
degree of polymerisation is achieved already within a
very short period of time which very quickly reaches a
maximum and can then be increased no more but develops
constantly. This means that there is a limit of
30 irradiation time for each significant intensity level of
radiation, within which a maximum polymerisation is
reached and no further increase of polymerisation can be
achieved by exceeding said limit through longer
irradiation.

35

In the example shown, a sharp increase of polymerisation occurs at an intensity level I_{UV} of 220 mW/cm² already after approximately 0.05 s and reaches its maximum value already after a duration of irradiation of 0.2 s -
5 0.3 s, in this example of approx. 80%. This shows that, at a sufficient intensity level of irradiation, a high-level maximum of polymerisation can be reached already after a short time of irradiation.

10 The surface heating of the fibrous material or of the mineral wool material at various intensity levels of the UV radiation is explained thereafter in more detail with reference to Fig. 3. The abscissa or X axis represents time t in seconds. The ordinate or Y axis shows the
15 temperature of the surface of the mineral wool material in °C. The horizontal dashed line at 20°C symbolises the ambient temperature as a reference value. In the present example starts from the assumption that the mineral wool material is provided at a temperature of approx. 20°C
20 before irradiation. The second horizontal dashed line at 200°C shows the upper limit of the thermal load on the organic constituents, such as the polymerisable substance or the binding agent of a usual mineral wool material at its surface. In the graph itself, the function of
25 temperature versus time is shown for two different intensity levels I_{UV1} and I_{UV2} , of which I_{UV1} is the smallest one in the case of the example 11 mW/cm², and I_{UV2} the highest intensity level in the case of the example 2000 mW/cm². It can be seen that the temperature
30 of the surface of the mineral wool material increases almost proportionally with time at a constant intensity level of the UV radiation. This linear temperature increase depends on the intensity level I_{UV} in a way that the gradient of this straight temperature line increases
35 with increasing intensity levels. If the mineral wool material is irradiated with high intensity levels of UV

radiation, the temperature limit of e.g. 200°C, at which damage occurs at the surface of the mineral wool material, is already reached after a relatively short time.

5

The new knowledge about these interrelationships can be put to use within the teaching of the present invention by reducing the irradiation periods when irradiating mineral wool materials to such an extent that the arc of transition from the steep ascent to the maximum of polymerisation may just be reached within this period. This allows the use of intensity levels which, although they would lead to damage at the surface after longer irradiation, do not cause damage at the surface at such short irradiation times and still allow the introduction of a sufficient intensity into the centre of the material in order to also allow a satisfactory polymerisation P within such irradiation period.

20 Together with the physical relations shown in Figs. 1 and 2, it becomes clear that there is a working range depending on the intensity level I_{UV} of UV radiation, the thickness d of the mineral wool product, the irradiation period t and the shown temperature limit, wherein a maximum and rapid polymerisation of substances in fibrous materials is possible without having to expect surface damage.

30 The mineral wool material may be present at a high temperature prior to irradiation due to preceding treatment steps. Further heating due to the irradiation may then cause the temperature limit to be exceeded relatively rapidly and thus cause thermal load of the organic constituents such as of the polymerisable substance or of the binding agent of a usual mineral wool material at the surface thereof. It may thus be envisaged

to cool the mineral wool material prior to irradiation e.g. by means of suitable devices as they are proposed e.g. in DE-A-44 06 863 or DE-A-44 10 020, respectively, whereby a rapid temperature rise due to irradiation to values beyond the temperature limit and consequently damage to the surface may be precluded.

The exemplary relationship according to the present invention between variations of intensity level on the one hand and polymerisation on the other hand, and the thickness d of a mineral wool product at bilateral UV irradiation is shown in Fig. 4. The abscissa shows in the same way as in Fig. 1 the thickness d of a mineral wool product of, in the case of the example, 12 cm. At the left ordinate, the intensity level I_{UV} from 0 to 100% and at the right ordinate the intensity level I_{UV} from 0 to 2000 mW/cm² is shown. In this, the intensity level of 100% corresponds to the 2000 mW/cm². Additionally the polymerisation P from 0 to 100% is shown at the right ordinate. Like in Fig. 1, the arrows $h\nu$ from the right and from the left symbolize the energy feed by irradiation with UV radiation. Curve 1 again shows the variation of the intensity level over the thickness d corresponding to Fig. 1. A curve 3 shows the variation of polymerisation P at irradiation from the left and a curve 4 shows the variation of polymerisation P at irradiation from the right. If irradiation takes place from both sides, the development of polymerisation P results from a superposition of curves 3 and 4, which is graphically represented by curve 2. In the exemplary case as shown, curves 3 and 4 are approximately mutual mirror images with the center of the mineral wool material as their axis, such that the curve 2 is also approximately symmetrical with the center portion.

In the following, curves 1 und 3 are first considered, on the basis of which the relation between the variation of the intensity level and polymerisation P shall be shown. As long as the intensity level I_{UV} , which sharply drops already after little penetration depth in curve 1, still exceeds a sufficiently high limit, an almost maximum polymerisation P takes place. If this limit is not reached, curve 3 drops as well and finally approaches zero. The same holds true for curve 4 at irradiation from the right. If the explanations given in respect of Figs. 1 to 3 are taken into consideration, it becomes clear that an almost uniformly good polymerisation can be achieved in the mineral wool material over its complete thickness at a sufficiently high starting intensity of the UV radiation at the surface of the mineral wool product, which then, typically, rapidly decreases, however not further than to a specific intensity limit at which polymerisation still noticeably occurs, and at a correspondingly chosen duration of irradiation. In this manner it would be possible to polymerise mineral wool materials across their entire thicknesses. However, it is also possible, for example by acting on the exposure time, to cause substances in these mineral wool materials to polymerise only in the vicinity of their surfaces and to cause no polymerisation or only a reduced polymerisation in their center portions.

Any unpolymerised parts of the substance within the fibrous material are exposed to ageing due their reacting with atmospheric oxygen and other reactants, whereby their reactivity is eliminated and the substance is made inert.

Fig. 5 shows a schematically simplified representation of an exemplary embodiment of a device according to the invention having linear focussing of the

UV radiation. A UV radiation source 10, preferably a linear UV radiation source, is located in the first focal point of a reflector 12 whose cross-section preferably has the form of a parabola or an ellipse. The UV radiation emitted by the UV radiation source 10 is focussed onto its second focal point 14 by means of the reflector 12. The second focal point 14 is located in the vicinity of the surface of the mineral wool material 16 to be irradiated or within the mineral wool material. The mineral wool material 16 may be provided as mineral wool material web and is, for example, transported on a conveyor device, not shown, underneath at least one UV radiation source 10 and/or above at least one UV radiation source 10, and thus swept by the linearly focussed beam 18 of a minimum width b approximately at right angles to the direction of transport.

Fig. 6 shows a schematically simplified representation of a device according to the invention producing a point-shaped focussed beam on the mineral wool material. The UV radiation source 10 is, in this case, point-shaped and arranged in the first focal point of an approximately semi-ellipsoidal reflector 12. By means of this semi-ellipsoidal reflector 12, UV radiation of the radiation source 10 is focussed in the second focal point 14. The second focal point 14, on the other hand, is located in the vicinity of the surface or within the mineral wool material 16, which may, for example, be formed as a three-dimensional shaped article, and whose surface contour is swept by the focussed UV beam 18 under an angle of penetration of approximately 90° to the planar normal of the respectively irradiated planar element. This can be accomplished by means of a suitable moving equipment for the UV radiation source 10, not shown, and/or by means of movable mirror units, also not shown.

The devices of Fig. 5 and 6 use finite focal points at the level of the mineral wool material to be treated. However, quite similar effects may also be obtained when
5 using thin beams with infinite focal points, i.e. parallel rays.

The oxygen content in the fibrous material during the treatment is to be reduced preferably to below 10% or
10 even more preferably to below 5% or even most preferably to below 1%, whereby the case in which the free radicals of the substance to be polymerised, which are produced by the UV radiation by means of the photo-initiators, are reacted with oxygen and a polymerisation at this position
15 is prevented may be avoided practically altogether.

In case of sufficient reduction of the atmospheric oxygen content in the fibrous material, a more complete polymerisation of the substance can thus be achieved. It
20 is a further advantage of the use of an inert atmosphere to reduce the possible formation of ozone since in a mostly inert atmosphere, oxygen molecules which might be partly split by the energy of the UV radiation to form oxygen radicals, and which might then form ozone are no
25 longer present.

The reduction of the oxygen content may be obtained in a simple manner by flushing the mineral wool material with another gas, for example nitrogen, or in a more
30 economic manner with carbon dioxide or water vapour.

Claims

1. Method for polymerising substances in fibrous materials, in particular binding agents in mineral wool materials for insulation purposes, wherein the fibrous material which has a given thickness (d) and which, in a given case, may be continuously moving in a line of production, is subjected to UV irradiation in order to polymerise the substance having the form of a prepolymer impregnating the fibres,
- characterised in that*
- irradiation of the material is carried out at a sufficiently high intensity level (I_{UV}) to bring about, at a maximum depth of intended polymerisation, a residual intensity exceeding such threshold value at which polymerisation of the selected binding agent under the influence of UV radiation is ensured within a given time limit, such time limit, however, precluding any undesirable degradation, due to the effects of the radiation, of organic substances in the portion at the surface of said material, and in the case of fibrous material continuously moving in a line of production being compatible with the velocity of the line; and
- in that* the duration (t) of irradiating a given surface unit of said material is kept within said time limit.
2. Method according to claim 1, *characterised in that* said intensity level (I_{UV}) at the surface of said

mineral wool material exceeds 500 mW/cm², preferably exceeds 1 W/cm², and in particular exceeds 2 W/cm².

- 5 3. Method according to claim 1 or 2, *characterised in that* the wavelength of the UV radiation utilised is in excess of 250 nm, preferably of 310 nm.
- 10 4. Method according any one of claims 1 to 3, *characterised in that* the duration (t) of irradiating said surface unit is shorter than ten seconds, preferably shorter than 1 s, and in particular shorter than 0.5 s.
- 15 5. Method according to any one of claims 1 to 4, *characterised in that* the surface of said mineral wool material is cooled prior to irradiation.
- 20 6. Method according to any one of claims 1 to 5, *characterised in that* the proportion of oxygen contained in the atmosphere inside said mineral wool material is reduced to less than 10 %, preferably to less than 5 %, in particular to less than 1 %.
- 25 7. Method according to any one of claims 1 to 6, *characterised in that* irradiation is applied to both major surfaces of said material.
- 30 8. Method according to claim 7, *characterised in that* both sides are irradiated concurrently.
9. Method according to claim 7 or 8, *characterised in that* a low-polymerisation zone is provided in the central region of said material.
- 35 10. Method according to any one of claims 1 to 9, *characterised in that* a mixture of substances

comprising an epoxy acrylate is used as the prepolymer for forming a binding agent.

- 5 11. Device for polymerising substances in fibrous materials, such as particularly a binding agent in a mineral wool material for insulation purposes, comprising at least one UV radiation source (10),
10 *characterised in that* means (12) are provided to focus the UV radiation into at least one focussed beam (18) of small width (b).
12. Device according to claim 11, *characterised in that* said focussed beam (18) is shaped to be linear, and is preferably kept stationary.
15
13. Device according to claim 11, *characterised in that* said focussed beam (18) has the shape of a point rapidly sweeping said mineral wool material (16) from one side to the other so as to irradiate preferably all of its surface area.
20
14. Device according to any one of claims 11 to 13, characterised that said focussed beam (18) has a focal point (14) located within said mineral wool material (16) to be treated.
25
15. Device according to any one of claims 11 to 14, *characterised in that* UV radiation sources (10) are positioned on both sides of the major surfaces of said mineral wool material (16).
30

Fig. 1

1 / 3

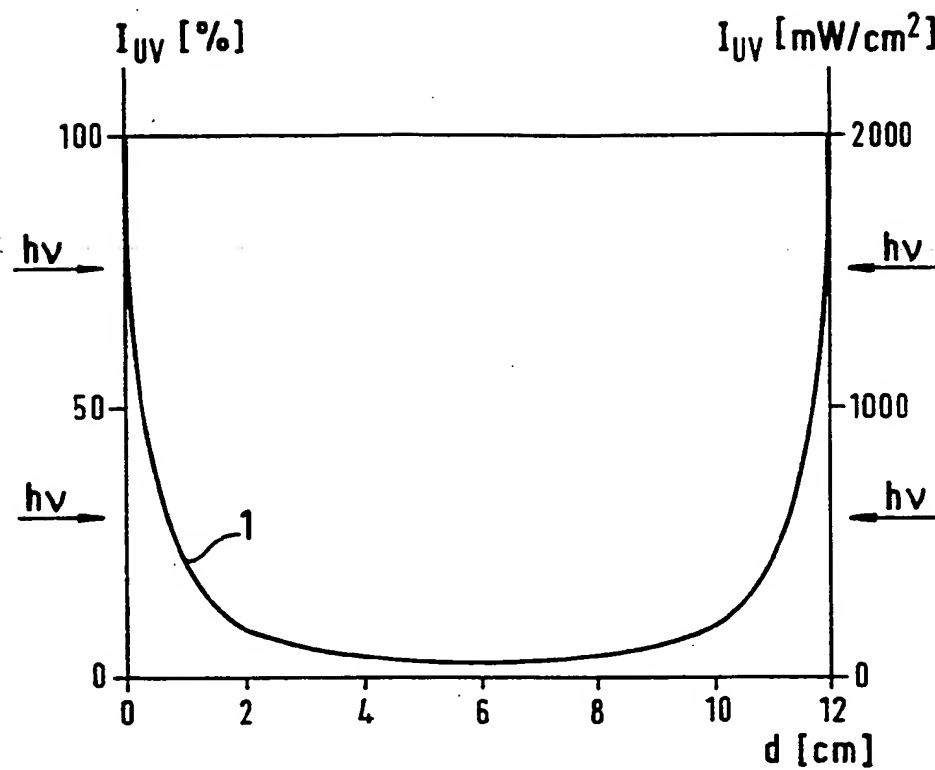


Fig. 2

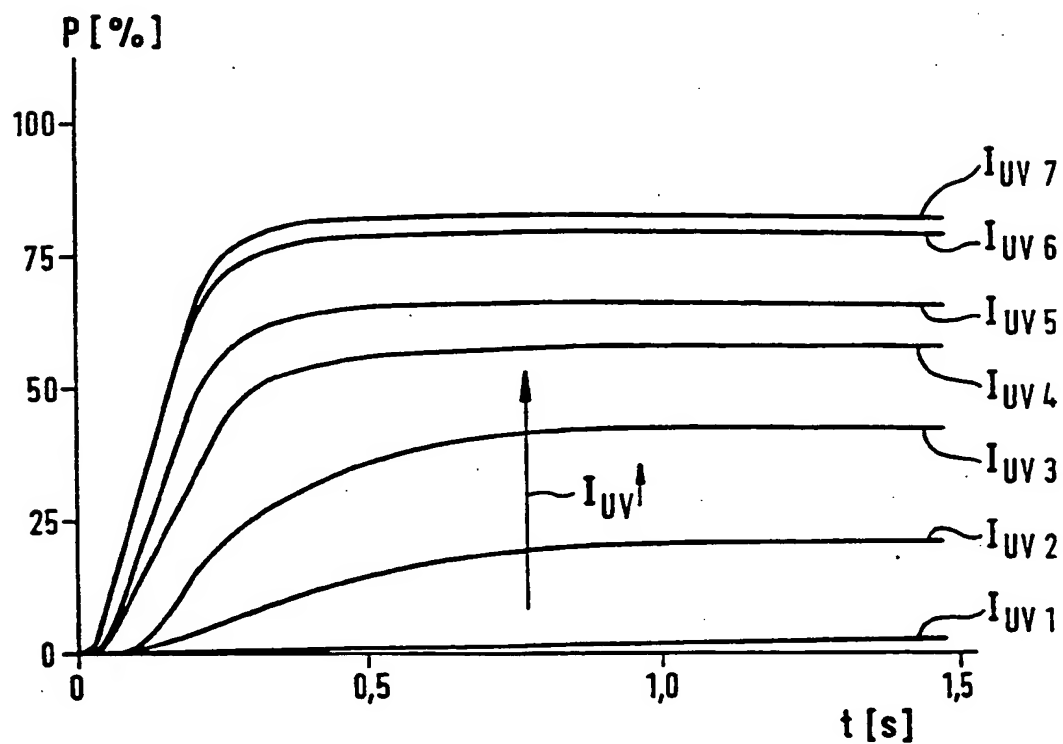


Fig. 3

2/3

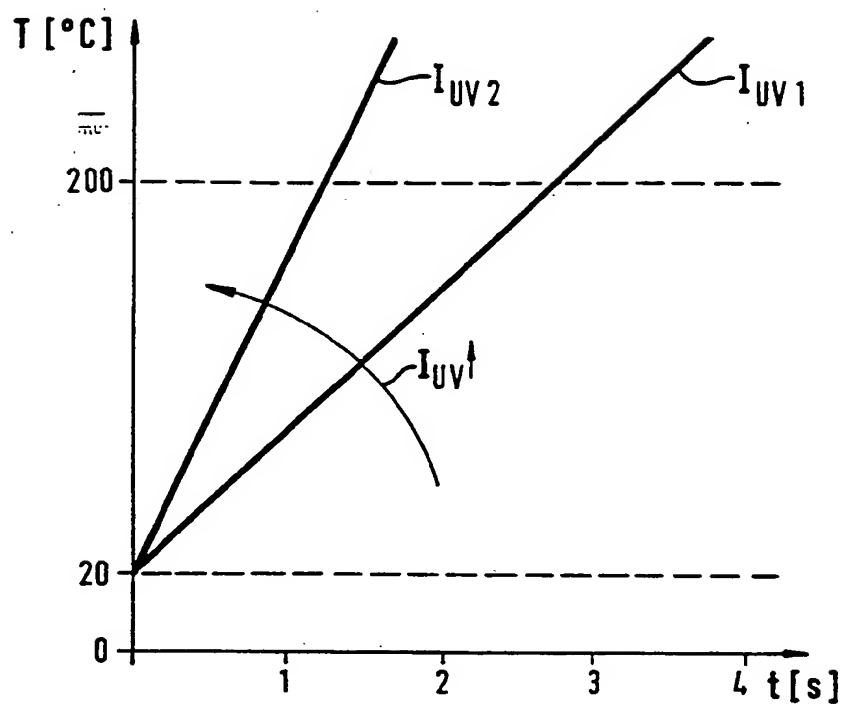
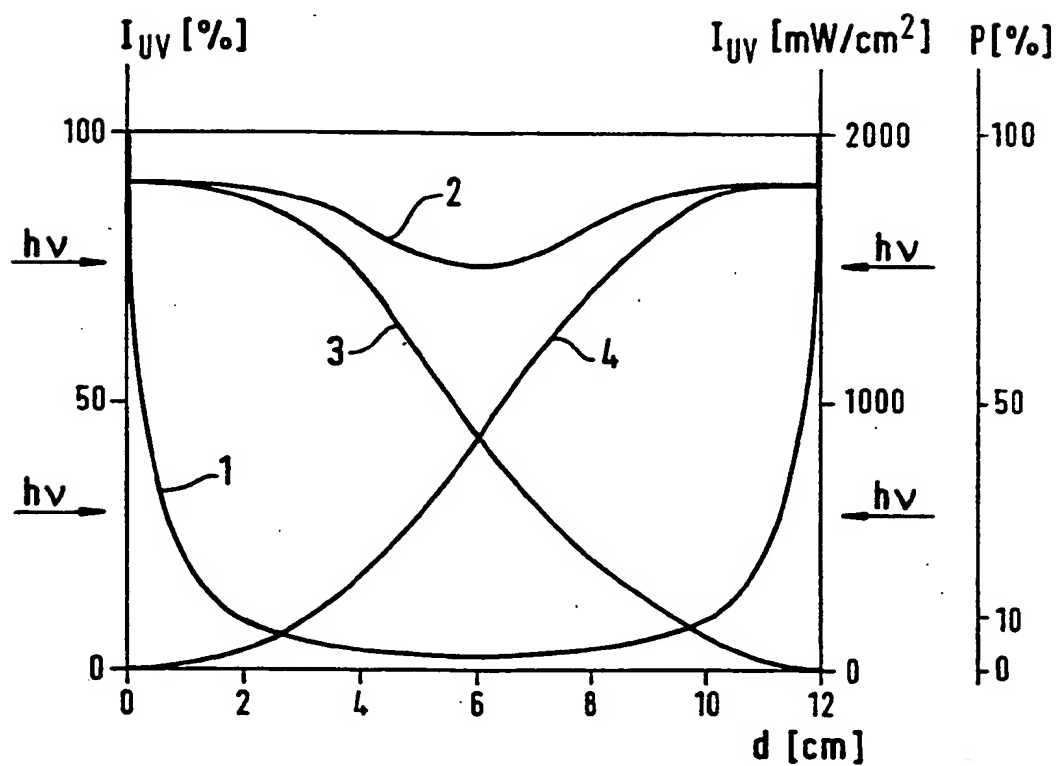


Fig. 4



3 / 3

Fig. 5

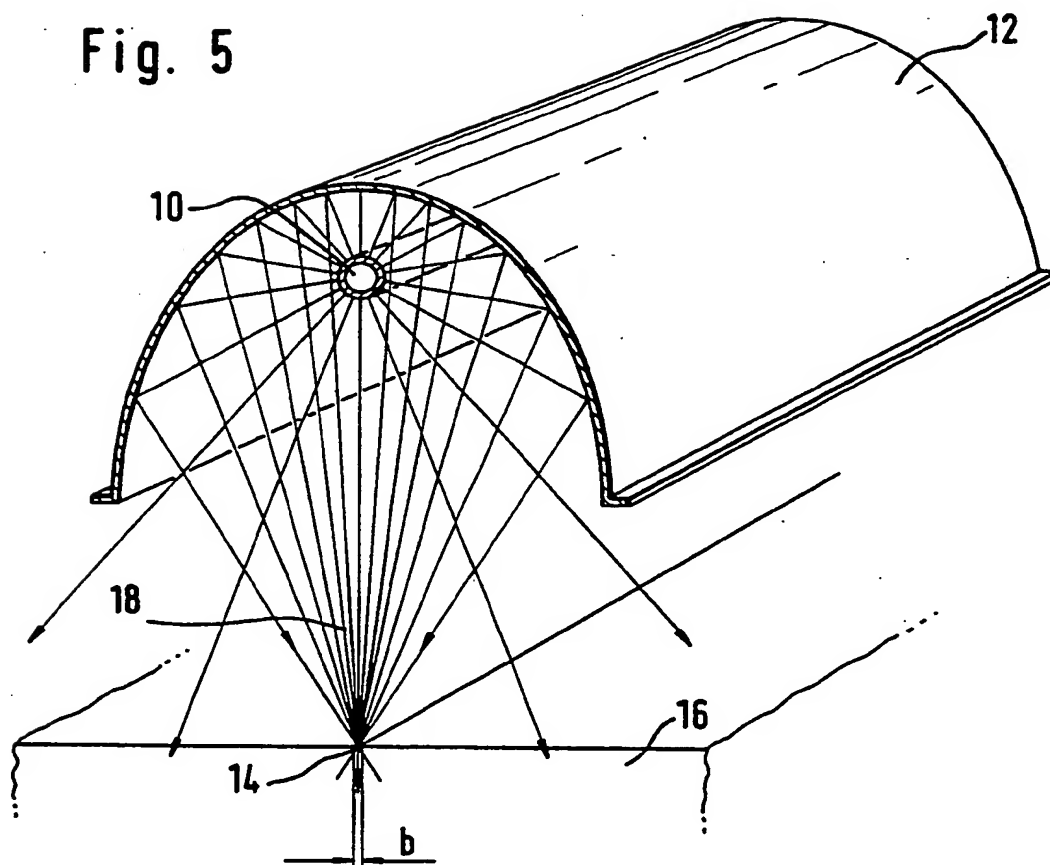
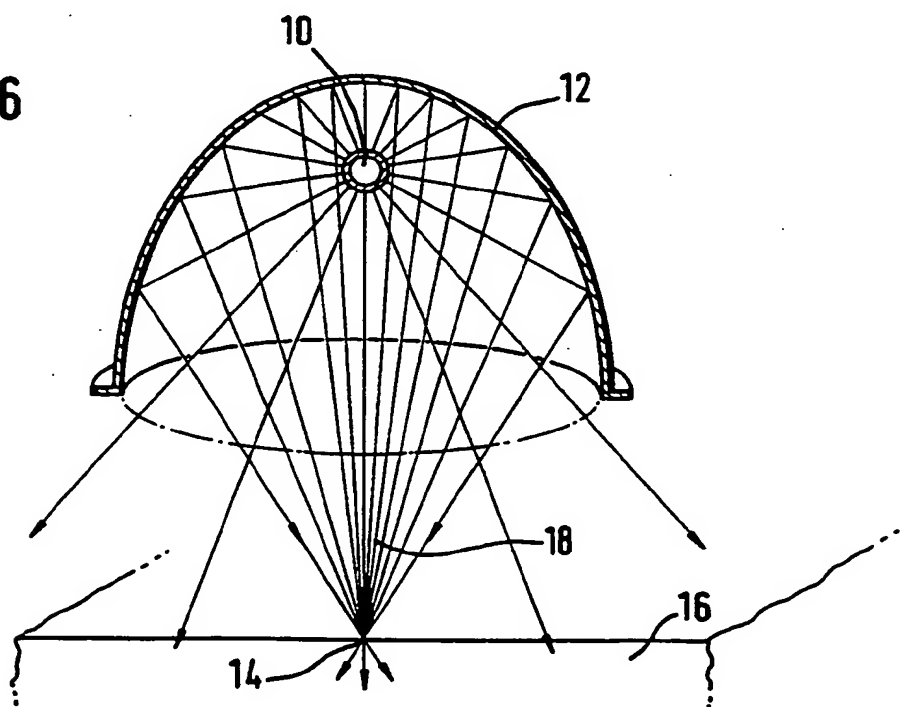


Fig. 6



INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 95/02325

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D04H1/64 D06M10/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D04H D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A,5 275 874 (K BRANDENBURG ET AL) 4 January 1994 cited in the application see the whole document ---	1,3
Y	MELLIAND TEXTILBERICHTE, vol. 74, no. 5, May 1993 HEIDELBERG DE, pages 428-434, M MARSMAN ET AL 'PIGMENTIERTE UV-HÄRTENDE BESCHICHTUNGEN FÜR TEXTILANWENDUNGEN' see page 432 ---	1,3
X	DE,A,29 37 081 (UNISEARCH LTD) 20 March 1980 see claims; tables 14,17 --- -/--	1,11

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of the actual completion of the international search

29 September 1995

Date of mailing of the international search report

20. 10. 95

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+ 31-70) 340-3016

Authorized officer

Heywood, C

International Application No
PCT/EP 95/02325

PCT/EP 95/02325

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE,A,21 24 691 (IMPERIAL CHEMICAL INDUSTRIES) 9 December 1971 see page 5; claims -----	11

BNSDOCID: <WO_____9535402A1_1_>

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 95/02325

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-5275874	04-01-94	NONE	
DE-A-2937081	20-03-80	AU-B- 536517	10-05-84
		AU-B- 5071079	20-03-80
		GB-A, B 2036039	25-06-80
		US-A- 4401688	30-08-83
DE-A-2124691	09-12-71	GB-A- 1349058	27-03-74